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## INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

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<b>(21) International Application Number:</b> PCT/GB91/00063 <b>(22) International Filing Date:</b> 16 January 1991 (16.01.91)  <b>(30) Priority data:</b> 9001108.1 18 January 1990 (18.01.90) GB  <b>(71) Applicant (for all designated States except US):</b> BRITISH TEXTILE TECHNOLOGY GROUP [GB/GB]; Shirley Towers, Didsbury Road, Didsbury, Manchester M20 8RX (GB).  <b>(72) Inventors; and</b> <b>(75) Inventors/Applicants (for US only) :</b> SAGAR, Brian [GB/GB]; 42 Moseley Road, Cheadle Hulme, Cheadle, Cheshire SK8 5HJ (GB). WALES, David [GB/GB]; 48 Browning Road, Reddish, Stockport, Cheshire SK5 6JN (GB). NELSON, Gordon [GB/GB]; 2 Edgeworth Drive, Manchester M14 6RU (GB).		<b>(74) Agents:</b> McNEIGHT, David, Leslie et al.; McNeight & Lawrence, Regent House, Heaton Lane, Stockport, Cheshire SK4 1BS (GB).  <b>(81) Designated States:</b> AT (European patent), AU, BE (European patent), CA, CH (European patent), DE (European patent), DK (European patent), ES (European patent), FR (European patent), GB (European patent), GR (European patent), IT (European patent), JP, LU (European patent), NL (European patent), PL, SE (European patent), SU, US.  <b>Published</b> <i>With international search report.</i> <i>Before the expiration of the time limit for amending the claims and to be republished in the event of the receipt of amendments.</i>
<b>(54) Title:</b> TREATING MATERIALS  <b>(57) Abstract</b>  Substances, e.g. insecticides, dyes, fragrances, are encapsulated in microcapsules of e.g. yeast and attached to materials e.g. of cotton or wool with a binder which does not impede the release of the substance from the microcapsule.		

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TREATING MATERIALS

This invention relates to treating materials, for example, textiles. Inter alia the invention relates to treating textiles with flame retardants, biocides, insecticides, odoriferous agents and dyes.

It is known that paper can be treated with dyes and odoriferous agents contained within microcapsules so that these can be released therefrom when the paper is subjected to compression, as when it is struck by a typewriter key hammer, for example, or compressed or abraded by a writing implement.

The choice of microcapsule has been recently expanded to include microbial cells such as those comprised by fungi, bacteria and algae as disclosed in European Patent Application 0,242,135, which describes adherence of the microcapsules to paper absent a binder or adhesive, although the intended contents for such microcapsules are limited to those classes which are already well known, viz, fragrances, dyes and biocides.

Moreover, the intended uses of such microcapsules are limited to those for which prior microcapsules have found applications. Such applications include those where the contents of the microcapsule are released

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continuously therefrom, as in coating paper to produce perfumed drawer liners, for example; or those applications where use of an article coated with or comprising encapsulated substances, for the purpose intended is destructive of further capacity of the article to provide the desired results. Thus, for example, normal use of non-carbon copy paper consumes its capacity for further normal use.

Thus prior microcapsules have found rather pedestrian application in either use once only articles, or in continuous release systems.

The present invention provides novel uses for micro-encapsulation.

According to the present invention there is provided a method for applying substances to materials comprising encapsulating the substances within microcapsules from which the substances can be released either by lesion of the microcapsules or by leaching of the substances therefrom and attaching the microcapsules to the materials with a binder which secures the microcapsules substantially against removal from the material during normal usage of the material but does not impede the release of the substances.

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The microcapsules may, for example, be a yeast such as Saccharomyces cerivisiae.

Alternatively said microcapsules may comprise starch or corn granules, or cellulosic fibres, such as those derived from wood pulp.

In the case of substances which may be released continuously from the microcapsules, the substances may be alkali-soluble biocides, odoriferous agents such as mint, clove or cedar wood oils, for example, dyes, or indeed any other appropriate material capable of being encapsulated by known means into said microcapsules.

Encapsulation of the aforementioned oils may be achieved by suspending dried yeast in a solution comprising an organic solvent and an oil and evaporating said solvent so that the oils enter by diffusion.

Of course, where desired, multiple oils may be added to said solvent, and the method according to the invention includes any suitable organic solvent-soluble substance or substances, in addition to the the afbresaid oils.

Said solvent may be ethanol.

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Where the substance is alkali soluble, it may be precipitated with acid within the microcapsule.

Said substance may, for example, be an organo-sulphur compound when it may be generally desired that it is not continuously released from the microcapsules but is released in response to a specific event.

The microcapsules may be bound to cotton in a moist-cure cross-linking process, using, for example, Fixapret CPN.

Preferably, however, an ethylene grafted vinyl acetate copolymer emulsion, such as Vinamul 6705 is used to bind the microcapsules to cotton.

When the textile is wool, a copolymer comprising adipic acid and diethylene triamine partially cross-linked with epichlorohydrin, such as Hercosett may be used as the binder. It will be appreciated, however, that a copolymer of methylmethacrylate and dimethylamino-ethylmethacrylate, for example, Dylan GRB, likewise treated with epichlorohydrin may also be used as a binder.

The invention will be further apparent from the following description, with reference to the several

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figures of the accompanying drawings, which illustrate the invention by way of examples only, with reference to the production of woollen carpets comprising encapsulated insecticides; and the production of fabrics comprising thiol compounds which in response to heat release malodorous gases which act as an indicator of a possible impending conflagration.

Of the drawings :-

Figure 1 shows schematically the precipitation of an alkali soluble biocide within yeast cells to yield a microcapsule containing biocide, prior to the application thereof, for example, to a woollen carpet;

Figure 2 likewise shows the microencapsulation of an organic solvent-soluble biocide within yeast cells to yield microcapsules containing biocide prior to the application thereof to textiles;

Figure 3 is from a scanning electron micrograph of cotton fabric with yeast bound by Vinamul 6705 thereto;

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Figure 4 is from a scanning electron micrograph of a cotton wool batt with yeast bound by Vinamul thereto; and

Figure 5 is from a scanning electron micrograph of a wool fibre with yeast bound thereto by Hercosett 125.

The present invention provides a method for applying substances to materials comprising encapsulating the substances within microcapsules from which the substances can be released either by lesion of the microcapsules or by leaching of the substances therefrom and attaching the microcapsules to the materials with a binder which secures the microcapsules substantially against removal from the material during normal usage of the material but does not impede the release of the substances.

Moth proofing agents, Mitin FF, Eulan WA and Perigen were dissolved in ethanol, and a dried yeast, Saccharomyces cerevisiae, added thereto. The solvent was evaporated from the resulting suspension, and the yeast cells comprising the thereby encapsulated insecticides were applied, using a spray technique in which the binder was vinamul 6705, to samples of woollen carpets.



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The Vinamul concentration was approximately 2% by weight of the carpet to which the insecticide containing-yeast cells were added.

The microencapsulated insecticide is, as a consequence of the method, contained within a micro-capsule and released therefrom in response to moth larvae attempting to devour the wool fibres to which the said micro-capsules are attached.

It will be appreciated that the amount of insecticide applied to the carpet is dependent upon the amount of insecticide encapsulated within the yeast cells, as well as on the number of yeast cells bound to the carpet. It may be preferred to encapsulate a relatively low level of insecticide within the cells but to bind a relatively high number of insecticide containing cells to the carpet. It is thought that moth larvae are attracted to yeast as a nutrient source. Encapsulating a small but fatal dose of biocide in each cell and attaching a large number of cells to the wool fibres will provide the capacity to kill large numbers of larvae without depleting the biocide level too much.

The nature of the binder is such that it does not disguise the nature of the microcapsule so that the moth larvae, for example continue to be attracted to the

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yeast cells rather than to the fibre, notwithstanding that these are embedded in a binder.

Figures 3 to 5 show the insecticide-containing microcapsules 9 bound with Vinamul 6705 (Figures 3 and 4) or Hercosett 125 (figure 5) to a cotton fibre 10 (Figure 3), a cotton batt 11 (Figure 4) or a wool fibre 12 (Figure 5).

The method, according to the invention provides that the microcapsule is not necessarily pre-formed, but may be formed in situ within the binder.

Thiol containing compounds which produce distinct odours when heated above temperatures encountered during processing and subsequent use can be encapsulated into an inert impervious polymer sheath during the production thereof, and subsequently incorporated into textiles, foams and plastics to act as a sensory early warning agent to indicate a possible conflagration. Vinamul 6705 or the "softer" variants such as Vinacryl 43309 and Vinacryl 43289 may comprise the polymer sheath.

It will be appreciated that it is not intended to limit the invention to the above example only, many variations, such as might readily occur to one skilled in the art, being possible, without departing from the scope thereof as defined by the appended claims.

For example, numerous end use applications of the method according to the invention will be apparent as a consequence of its disclosure.

Although the examples describe application of the microencapsulated substances to substrata by a spraying process, it will be appreciated that a padding process, or indeed a coating, or exhaustion process may be sometimes used with advantage depending on the desired result.

The amount of binder in which the microcapsules are dispersed may be varied so as to affect the distribution of capsules applied to a particular textile. Moreover, it may be desired to alter the accessibility of said capsules so as to expose greater or lesser regions of these to the event which causes release of the substances contained therein.

It will be obvious that the various binders must be treated having regard to the nature of the capsules dispersed therein, so that, for example, treatments which are not detrimental to the binder but which are harmful to the microcapsules and/or contents thereof and vice versa are not used.

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The invention is not limited to the examples of the binders described herein, and includes many other suitable polymers, such as polyurethanes and derivatives thereof.

It may be desired to use as a binder, a polymer which when polymerised comprises pores so that volatile substances encapsulated in microcapsules which are completely submerged within the binder are still able to permeate to the environment.

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CLAIMS

1. A method for applying a substance to a material comprising encapsulating the substance within microcapsules from which the substance can be released and attaching the microcapsules to the material with a binder which does not prevent release of the substance.
2. A method according to claim 1, in which the microcapsules comprise a yeast.
3. A method according to claim 2, in which the yeast comprises Saccharomyces cerivisiae.
4. A method according to claim 1, in which the microcapsules comprise starch.
5. A method according to claim 1, in which the microcapsules comprise corn granules.
6. A method according to claim 1, in which the microcapsules comprise cellulosic fibres.
7. A method according to claim 6, in which the cellulosic fibres are from wood pulp.

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8. A method according to claim 1, in which the microcapsules are bound to the material by a moist-cure cross-linking process.

9. A method according to claim 1, in which the microcapsules are bound to the material using an ethylene grafted vinyl acetate copolymer emulsion.

10. A method according to claim 1, in which the microcapsules are bound to the material using a copolymer comprising adipic acid and diethylene triamine partially cross-linked with epichlorohydrin.

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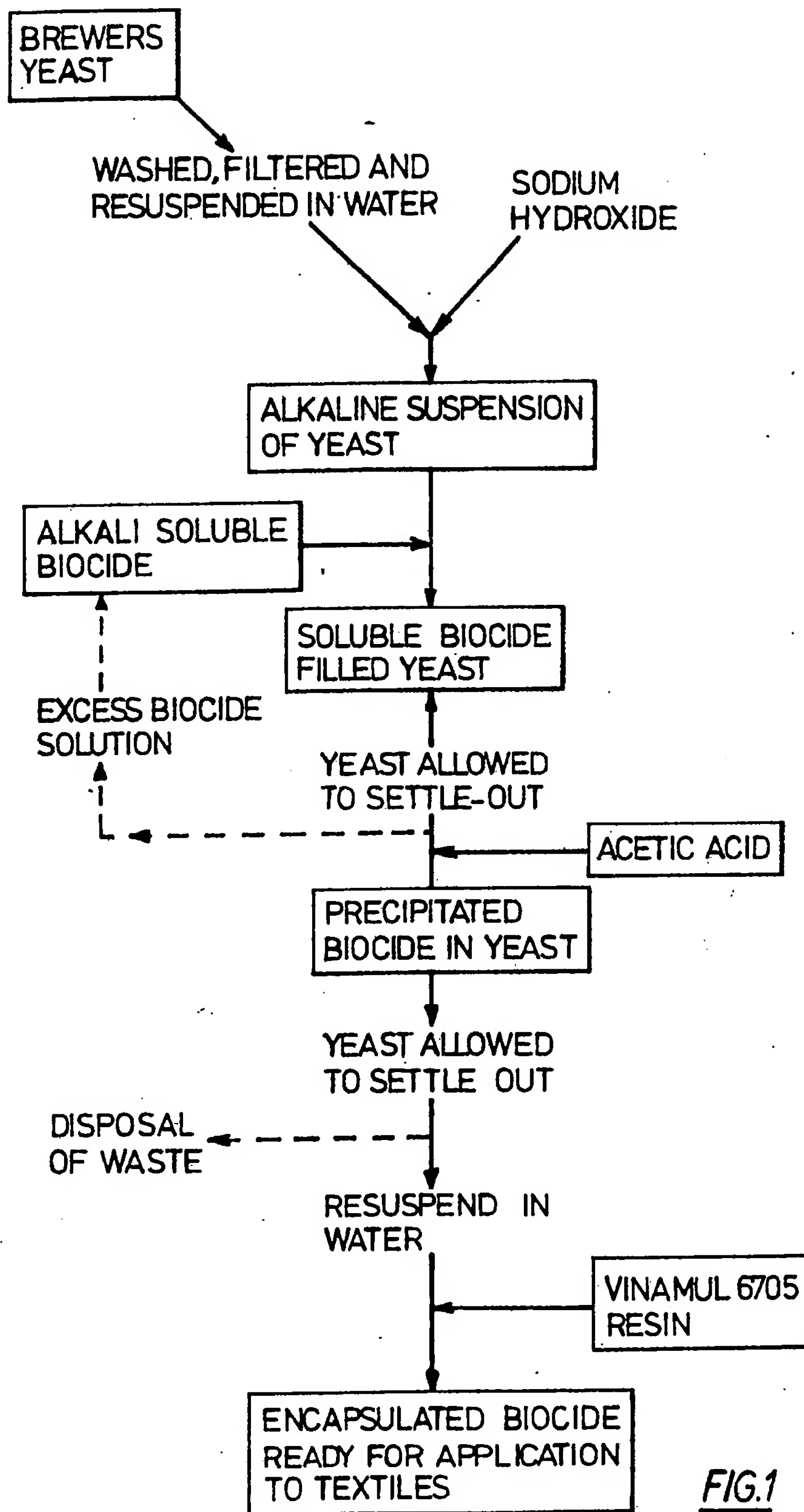
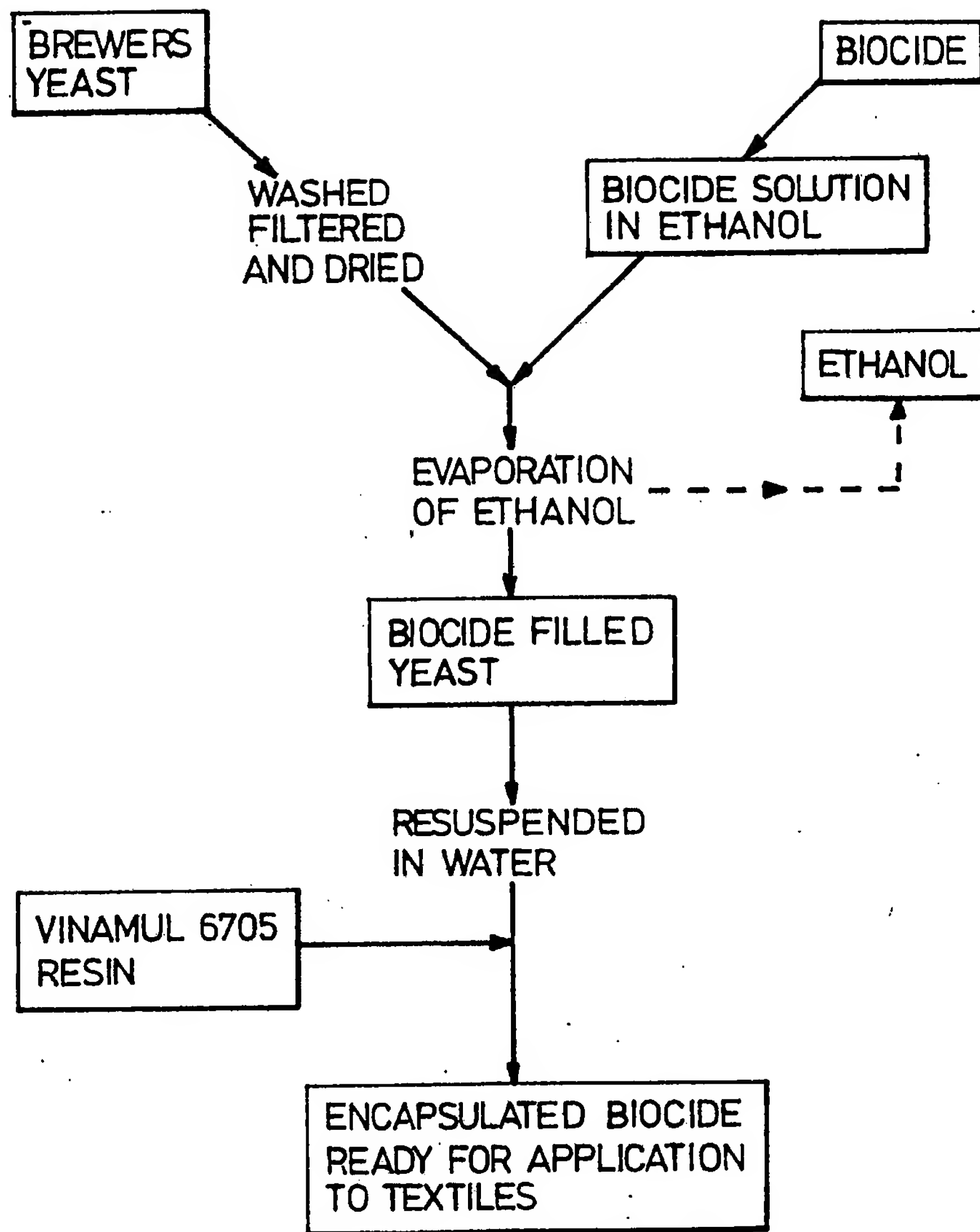


FIG.1

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FIG.2



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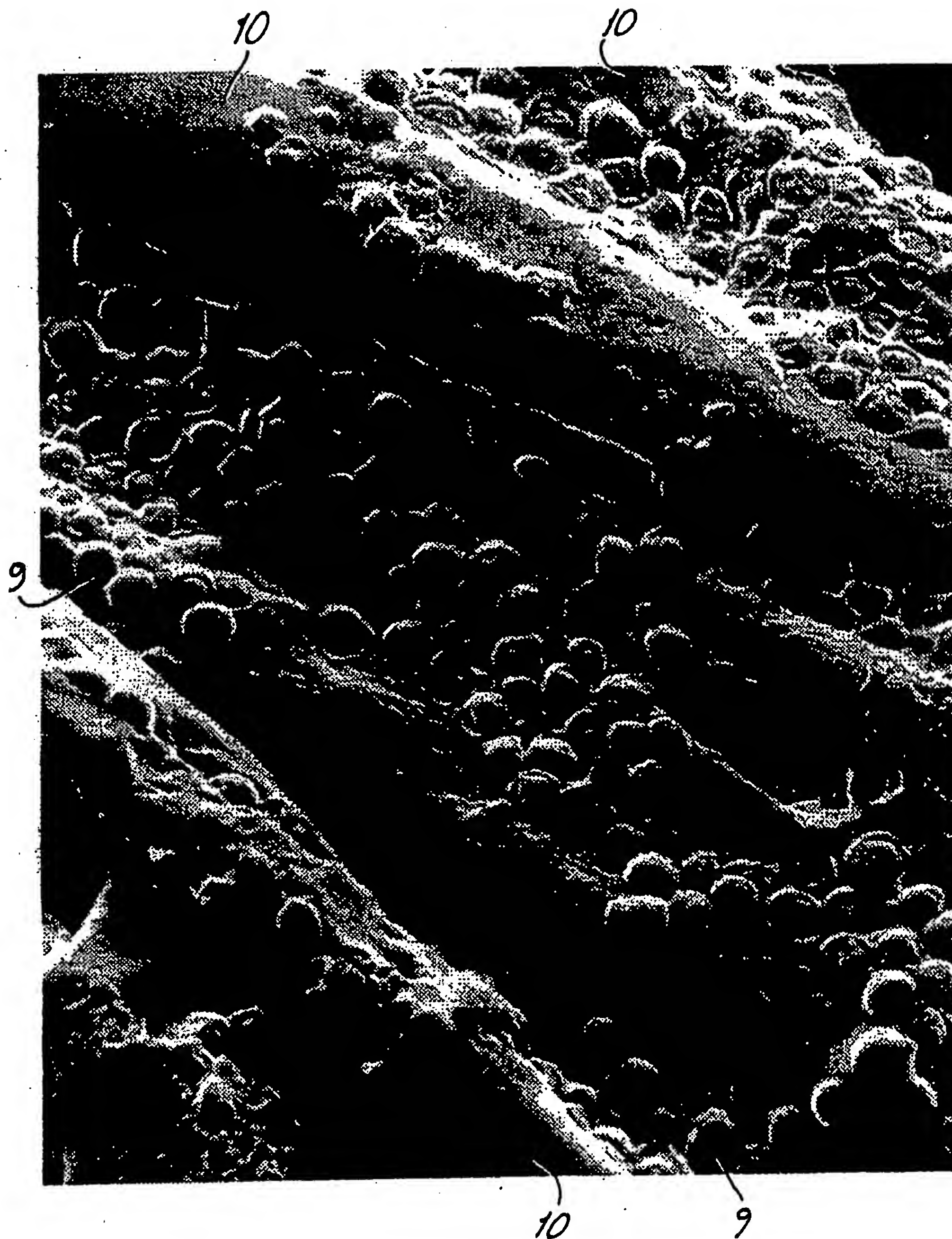


FIG.3

SUBSTITUTE SHEET

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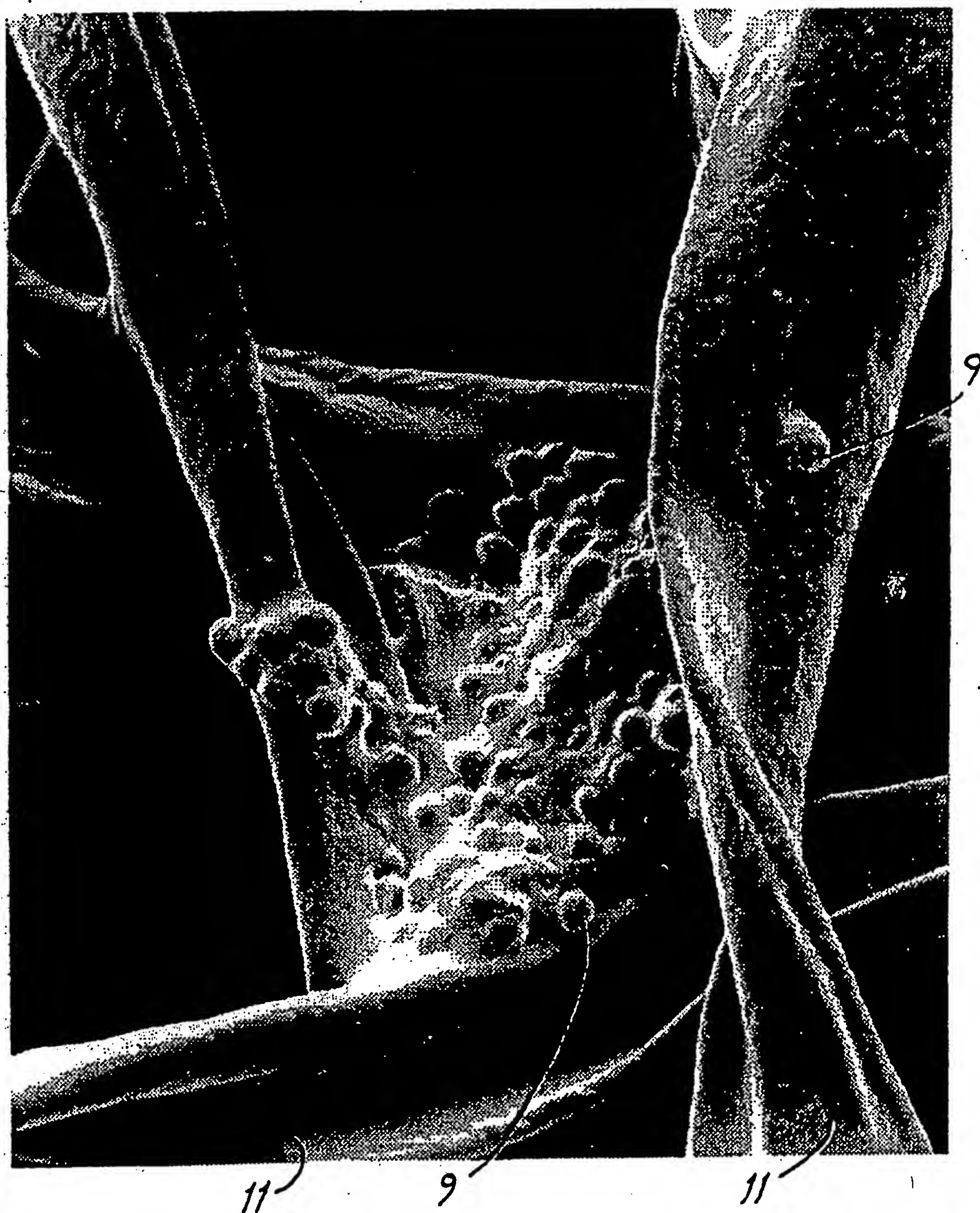


FIG.4

SUBSTITUTE SHEET

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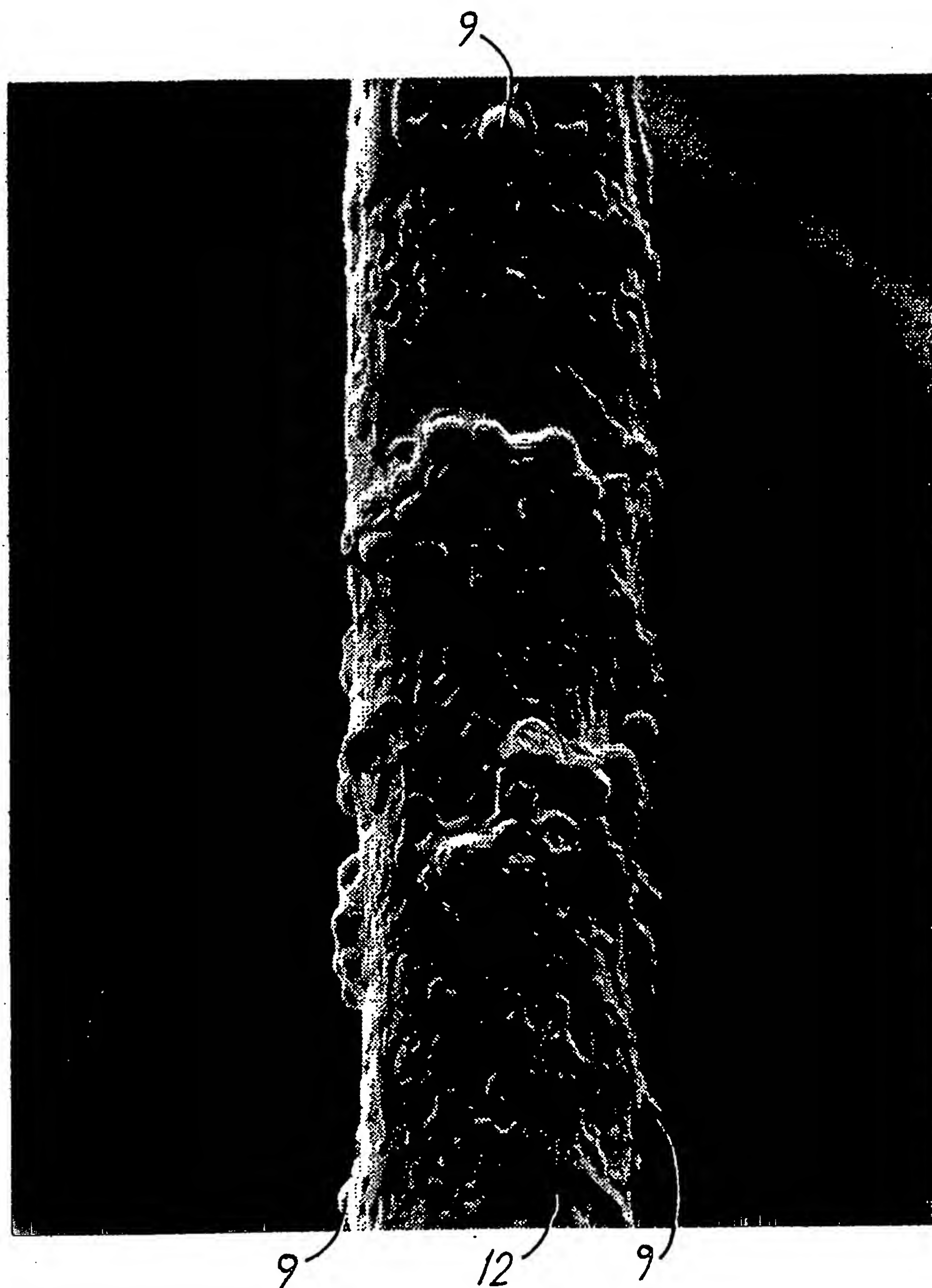
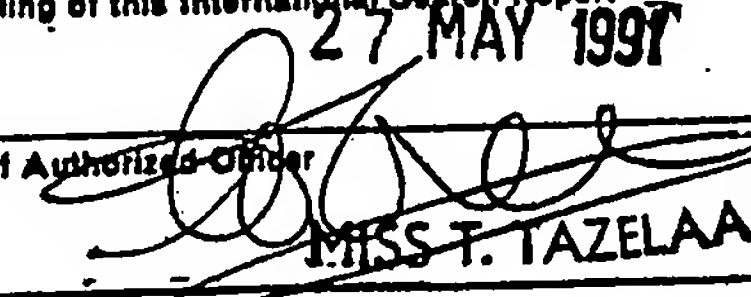


FIG.5

# INTERNATIONAL SEARCH REPORT

PCT/GB 91/00063

International Application No

<b>I. CLASSIFICATION OF SUBJECT MATTER</b> (if several classification symbols apply, indicate all) * According to International Patent Classification (IPC) or to both National Classification and IPC		
IPC <sup>5</sup> :            D 06 M 23/12		
<b>II. FIELDS SEARCHED</b>		
Minimum Documentation Searched <sup>7</sup>		
Classification System	Classification Symbols	
IPC <sup>5</sup>	D 06 M	
Documentation Searched other than Minimum Documentation to the extent that such Documents are included in the Fields Searched *		
<b>III. DOCUMENTS CONSIDERED TO BE RELEVANT</b> *		
Category *	Citation of Document, <sup>11</sup> with indication, where appropriate, of the relevant passages <sup>12</sup>	Relevant to Claim No. <sup>13</sup>
X	US, A, 4696863 (TOSHIHIKO MATSUSHITA) 29 September 1987 see column 2, lines 26-37; column 30, lines 40-59; column 4, line 26; column 8, lines 6-50 ---	1, 2, 3
X	EP, A, 0328937 (KANEBO LTD.) 23 August 1989 see page 2, lines 36-55; page 3, lines 1-14, 37-44 ---	1
X	US, A, 4528226 (N.P. SWEENEY) 9 July 1985 see column 3, lines 24-48; column 4, lines 31-47 ---	1
X	FR, A, 2005271 (THE NATIONAL CASH REGISTER CO.) 12 December 1969 see page 2, lines 9-21; page 21, lines 20-22 ---	1, 8, 9
<div style="display: flex; justify-content: space-between;"> <div style="width: 45%;"> <p>* Special categories of cited documents: <sup>10</sup></p> <p>"A" document defining the general state of the art which is not considered to be of particular relevance</p> <p>"E" earlier document but published on or after the international filing date</p> <p>"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)</p> <p>"O" document referring to an oral disclosure, use, exhibition or other means</p> <p>"P" document published prior to the international filing date but later than the priority date claimed</p> </div> <div style="width: 45%;"> <p>"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention</p> <p>"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step</p> <p>"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art</p> <p>"A" document member of the same patent family</p> </div> </div>		
<b>IV. CERTIFICATION</b>		
Date of the Actual Completion of the International Search <div style="text-align: center; font-size: 1.2em;">16th April 1991</div>		Date of Mailing of this International Search Report <div style="text-align: center; font-size: 1.2em;">27 MAY 1991</div>
International Searching Authority <div style="text-align: center; font-weight: bold;">EUROPEAN PATENT OFFICE</div>		Signature of Authorized Officer <div style="text-align: center;">   <b>MISS T. TAZELAAR</b> </div>

III. DOCUMENTS CONSIDERED TO BE RELEVANT (CONTINUED FROM THE SECOND SHEET)		
Category *	Citation of Document, <sup>11</sup> with indication, where appropriate, of the relevant passages	Relevant to Claim No.
X	EP, A, 0326444 (LAINIERE DE PICARDIE) 2 August 1989 see column 4, lines 41-61; column 5, lines 7-10; column 5, lines 50-59  ---	1,8,9
X	US, A, 3859151 (D.N. VINCENT) 7 January 1975 see column 3, lines 24-29,62-66  ---	1,4
A	EP, A, 0242135 (AD2 LIMITED) 21 October 1987 see page 2, lines 37-38; page 3, lines 1-13; page 4, lines 1-6 cited in the application  -----	1



# ANNEX TO THE INTERNATIONAL SEARCH REPORT ON INTERNATIONAL PATENT APPLICATION NO.

GB 9100063  
SA 43515

This annex lists the patent family members relating to the patent documents cited in the above-mentioned international search report.  
The members are as contained in the European Patent Office EDP file on 15/05/91  
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		JP-A- 2127572	16-05-90
		US-A- 4990392	05-02-91
US-A- 3859151	07-01-75	None	
EP-A- 0242135	21-10-87	None	

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For more details about this annex : see Official Journal of the European Patent Office, No. 12/82

insecticide within the cells but to bind a relatively high number of insecticide containing cells to the carpet. It is thought that moth larvae are attracted to yeast as a nutrient source. Encapsulating a small but fatal dose of biocide in each cell and attaching a large number of cells to the wool fibres will provide the capacity to kill large numbers of larvae without depleting the biocide level too much.

The nature of the binder is such that it does not disguise the nature of the microcapsule so that the moth larvae, for example continue to be attracted to the yeast cells rather than to the fibre, notwithstanding that these are embedded in a binder.

Figures 3 to 5 show the insecticide-containing microcapsules 9 bound with Vinamul 6705 (Figures 3 and 4) or Hercosett 125 (figure 5) to a cotton fibre 10 (Figure 3), a cotton batt 11 (Figure 4) or a wool fibre 12 (Figure 5).

The method, according to the invention provides that the microcapsule is not necessarily pre-formed, but may be formed in situ within the binder.

It will be appreciated that it is not intended to limit the invention to the above example only, many variations, such as might readily occur to one skilled in the art, being possible, without departing from the scope thereof as defined by the appended claims.

For example, numerous end use applications of the method according to the invention will be apparent as a consequence of its disclosure.

Although the examples describe application of the microencapsulated substances to substrata by a spraying process, it will be appreciated that a padding process, or indeed a coating, or exhaustion process may be sometimes used with advantage depending on the desired result.

The amount of binder in which the microcapsules are dispersed may be varied so as to affect the distribution of capsules applied to a particular textile. Moreover, it may be desired to alter the accessibility of said capsules so as to expose greater or lesser regions of these to the event which causes release of the substances contained therein.

It will be obvious that the various binders must be treated having regard to the nature of the capsules dispersed therein, so that, for example, treatments which are not detrimental to the binder but which are harmful to the microcapsules and/or contents thereof and vice versa are not used.

The invention is not limited to the examples of the binders described herein, and includes many other suitable polymers, such as polyurethanes and derivatives thereof.

#### Claims

1. A method for applying an insecticide to a fibrous textile material characterised by encapsulating the

insecticide within microcapsules of a substance which is a food to which insects or their larvae are attracted and attaching the microcapsules to the fibres of the material with a binder which does not impede the release of the insecticide when the microcapsules are devoured.

2. A method according to claim 1, characterised in that the substance is yeast.
3. A method according to claim 2, characterised in that the yeast comprises Saccharomyces cerevisiae.
4. A method according to any one of claims 1 to 3, characterised in that large numbers of microcapsules each containing a small but fatal dose of insecticide are applied.
5. A method according to claim 1, characterised in that the microcapsules comprise starch or corn granules.
6. A method according to claim 1, characterised in that the microcapsules comprise cellulosic fibres.
7. A method according to claim 6, characterised in that the cellulosic fibres are from wood pulp.
8. A method according to any one of claims 1 to 7, characterised in that the microcapsules are bound to the material by a moist-cure cross-linking process.
9. A method according to any one of claims 1 to 8, characterised in that the microcapsules are bound to the material using an ethylene grafted vinyl acetate copolymer emulsion.
10. A method according to any one of claims 1 to 9, characterised in that the microcapsules are bound to the material using a copolymer comprising adipic acid and diethylene triamine partially cross-linked with epichlorohydrin.

#### Patentansprüche

1. Verfahren zur Aufbringung eines Insektizids auf ein faserförmiges Textilmaterial, dadurch gekennzeichnet, daß das Insektizid in Mikrokapseln einer Substanz verkapselt wird, welche ein Nahrungsmittel ist, von dem Insekten oder deren Larven angezogen werden, und die Mikrokapseln an den Fasern des Materials mit einem Bindemittel befestigt werden, das die Freisetzung des Insektizids nicht behindert, wenn die Mikrokapseln gefressen werden.